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FILE 'HOME' ENTERED AT 10:45:43 ON 01 DEC 2006

=> file casreact
COST IN U.S. DOLLARS

FULL ESTIMATED COST

SINCE FILE ENTRY	TOTAL SESSION
0.21	0.21

FILE 'CASREACT' ENTERED AT 10:46:04 ON 01 DEC 2006
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FILE CONTENT:1840 - 27 Nov 2006 VOL 145 ISS 22

New CAS Information Use Policies, enter HELP USAGETERMS for details.

*
* CASREACT now has more than 10 million reactions
*

Some CASREACT records are derived from the ZIC/VINITI database (1974-1991) provided by InfoChem, INPI data prior to 1986, and Biotransformations database compiled under the direction of Professor Dr. Klaus Kieslich.

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> Uploading C:\Program Files\Stnexp\Queries\10735408.str

L1 STRUCTURE UPLOADED

=> d l1
L1 HAS NO ANSWERS
L1 STR
*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

Structure attributes must be viewed using STN Express query preparation.

=> s 11 sss sam
SAMPLE SEARCH INITIATED 10:46:55 FILE 'CASREACT'
SCREENING COMPLETE - 1601 REACTIONS TO VERIFY FROM 114 DOCUMENTS

100.0% DONE 1601 VERIFIED 7 HIT RXNS 4 DOCS
SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE **COMPLETE**
BATCH **COMPLETE**
PROJECTED VERIFICATIONS: 29623 TO 34417
PROJECTED ANSWERS: 4 TO 199

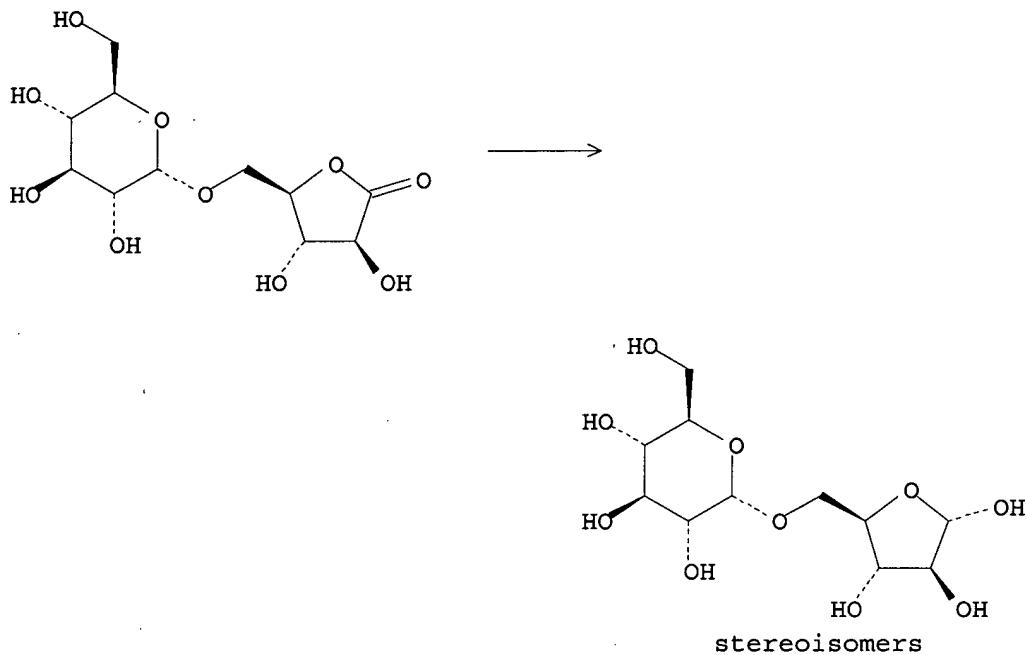
L2 4 SEA SSS SAM L1 (7 REACTIONS)

=> d scan

L2 4 ANSWERS CASREACT COPYRIGHT 2006 ACS on STN

TI Disaccharide building blocks from isomaltulose: glucosyl- α (1-5)-D-arabinonic acid and ensuing products

RX(3) OF 6



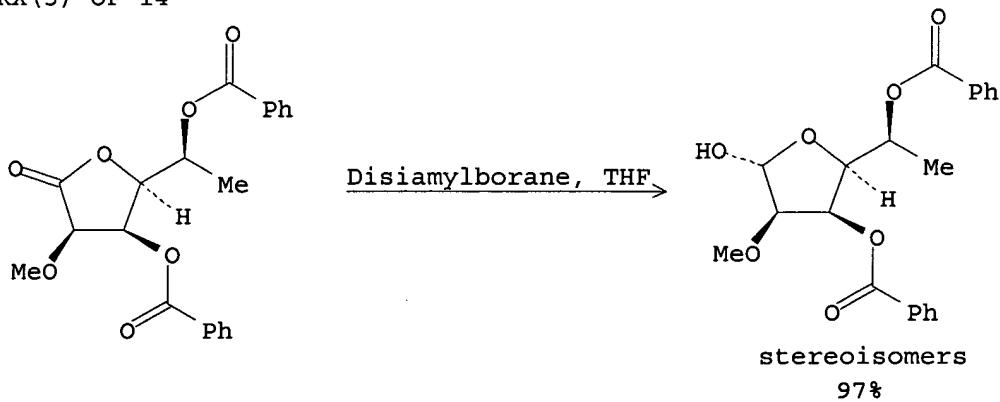
NOTE: 2 steps

HOW MANY MORE ANSWERS DO YOU WISH TO SCAN? (1):1

L2 4 ANSWERS CASREACT COPYRIGHT 2006 ACS on STN

TI The use of aldonolactones for the synthesis of 2-O-methyl-L-rhamnose and 2-O-methyl-D-mannose

RX(5) OF 14

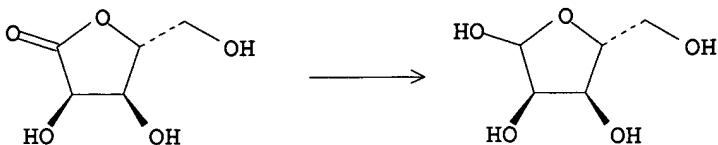


HOW MANY MORE ANSWERS DO YOU WISH TO SCAN? (1):1

L2 4 ANSWERS CASREACT COPYRIGHT 2006 ACS on STN

TI Preparation of D-ribose from D-ribono- γ -lactone

RX(1) OF 1



HOW MANY MORE ANSWERS DO YOU WISH TO SCAN? (1):end

=> s 11 sss full
FULL SEARCH INITIATED 10:50:58 FILE 'CASREACT'
SCREENING COMPLETE - 28991 REACTIONS TO VERIFY FROM 2093 DOCUMENTS

100.0% DONE 28991 VERIFIED 70 HIT RXNS 26 DOCS
SEARCH TIME: 00.00.03

L3 26 SEA SSS FUL L1 (70 REACTIONS)

=> s 13 and (NaHTe or SmI2)
32 NAHTE

924 SmI2

L4 0 L3 AND (NAHTE OR SmI2)

=> s 13 and tellurohydride
0 TELLUROHYDRIDE

L5 0 L3 AND TELLUROHYDRIDE

=> s 13 and (samarium(a)iodide)

2406 SAMARIUM

3 SAMARIUMS

2406 SAMARIUM

(SAMARIUM OR SAMARIUMS)

24362 IODIDE

4436 IODIDES

25885 IODIDE

(IODIDE OR IODIDES)

598 SAMARIUM(A)IODIDE

L6 0 L3 AND (SAMARIUM(A)IODIDE)

=> s 13 and (hydrogen(a)palladium(a)phosphine)

34904 HYDROGEN

651 HYDROGENS

35338 HYDROGEN

(HYDROGEN OR HYDROGENS)

23706 PALLADIUM

6 PALLADIUMS

23706 PALLADIUM

(PALLADIUM OR PALLADIUMS)

15380 PHOSPHINE

4762 PHOSPHINES

16636 PHOSPHINE

(PHOSPHINE OR PHOSPHINES)

0 HYDROGEN(A)PALLADIUM(A)PHOSPHINE

L7 0 L3 AND (HYDROGEN(A)PALLADIUM(A)PHOSPHINE)

=> s 13 and (hydrogen(a)catalyst)

34904 HYDROGEN
651 HYDROGENS
35338 HYDROGEN
(HYDROGEN OR HYDROGENS)

84032 CATALYST
78456 CATALYSTS
100975 CATALYST
(CATALYST OR CATALYSTS)

208 HYDROGEN (A) CATALYST

L8 0 L3 AND (HYDROGEN (A) CATALYST)

=> s 13 and (aluminum (w) t-butoxy (w) hydride)

12332 ALUMINUM
14 ALUMINUMS
12335 ALUMINUM
(ALUMINUM OR ALUMINUMS)

11297 T
1566 BUTOXY
20 T-BUTOXY
(T (W) BUTOXY)

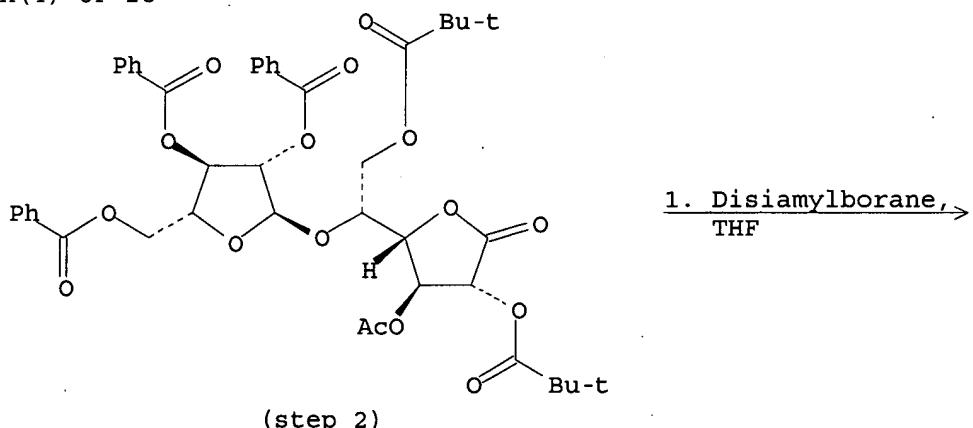
17641 HYDRIDE
1702 HYDRIDES
18107 HYDRIDE
(HYDRIDE OR HYDRIDES)

L9 0 ALUMINUM (W) T-BUTOXY (W) HYDRIDE
0 L3 AND (ALUMINUM (W) T-BUTOXY (W) HYDRIDE)

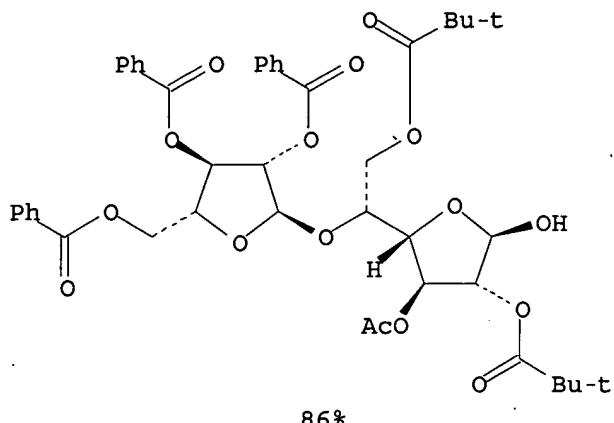
=> d 13 FCRDREF

L3 ANSWER 1 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

RX (4) OF 28



RX(4) OF 28

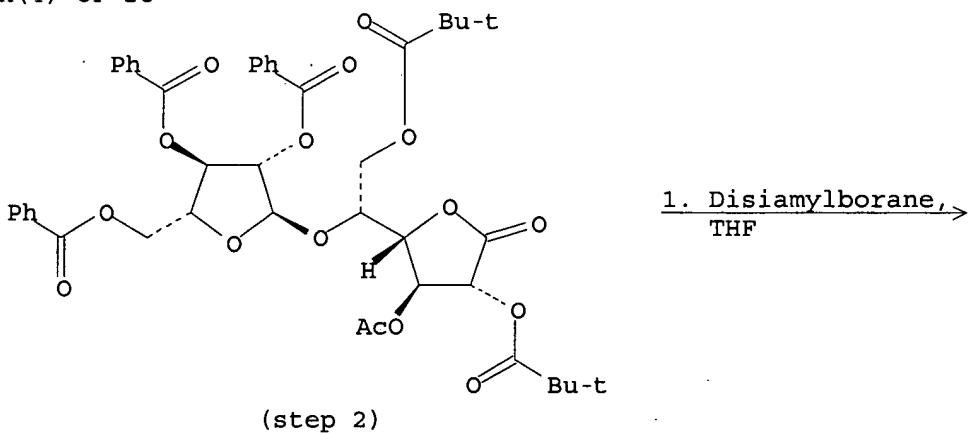


REF: Canadian Journal of Chemistry, 84(4), 486-491; 2006
NOTE: stereoselective
CON: STAGE(1) room temperature -> 0 deg C
STAGE(2) 0 deg C; 22 hours, room temperature

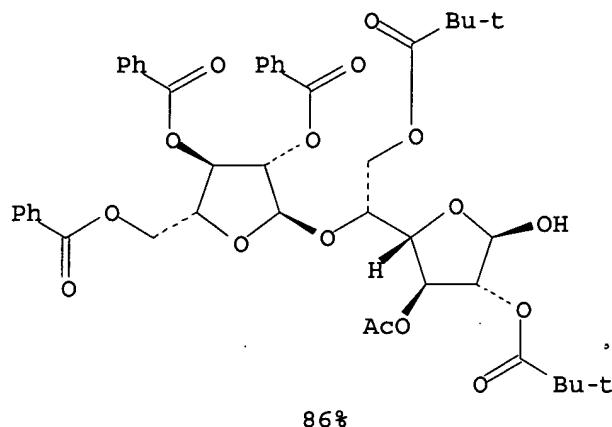
=> d 13 1-26 FCRDREF

L3 ANSWER 1 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

RX(4) OF 28



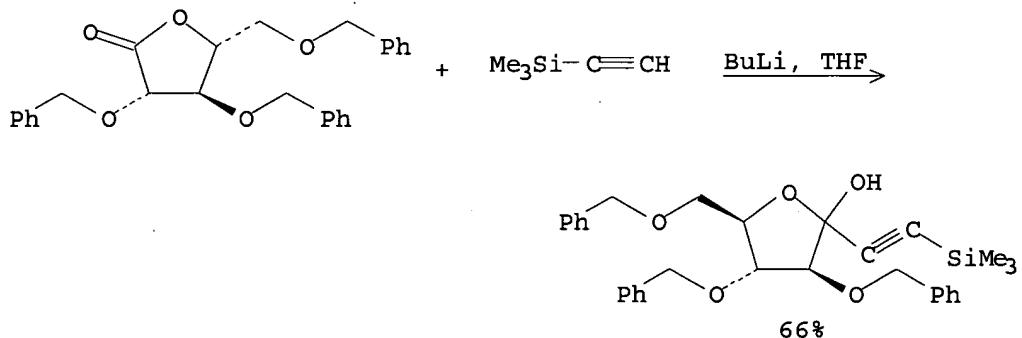
RX(4) OF 28



REF: Canadian Journal of Chemistry, 84(4), 486-491; 2006
NOTE: stereoselective
CON: STAGE(1) room temperature -> 0 deg C
STAGE(2) 0 deg C; 22 hours, room temperature

L3 ANSWER 2 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

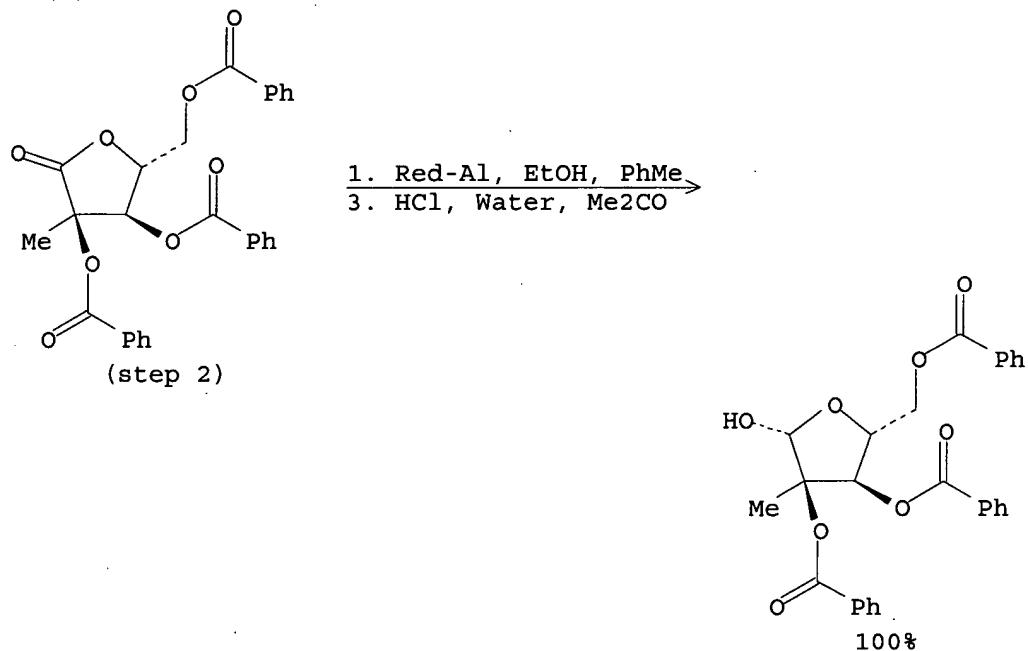
RX(11) OF 41



REF: Tetrahedron Letters, 46(18), 3249-3252; 2005
NOTE: stereoselective
CON: -78 deg C

L3 ANSWER 3 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

RX(3) OF 115

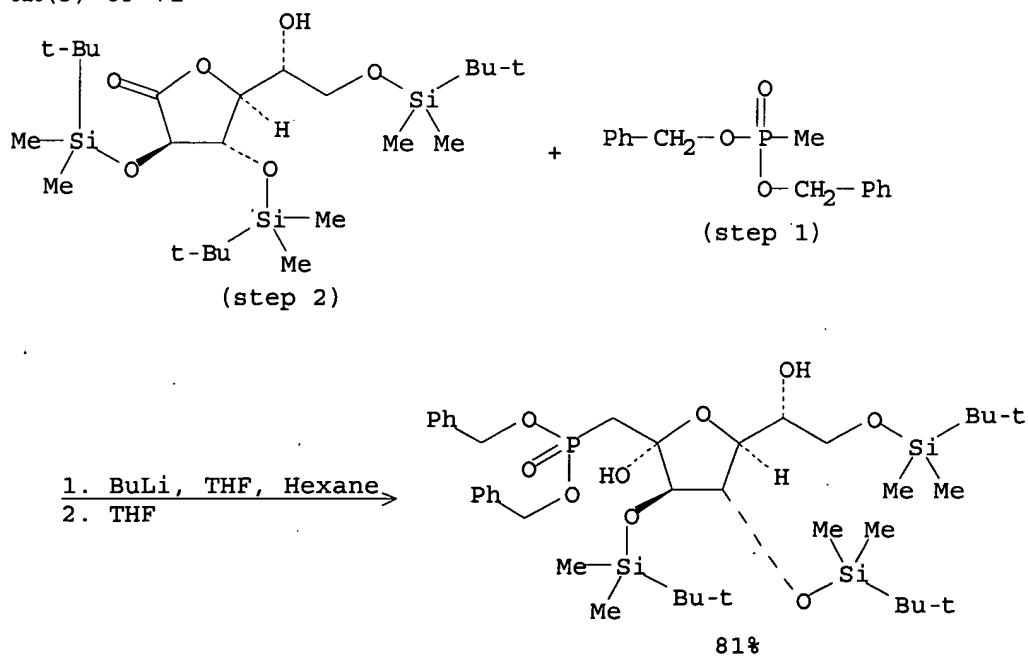


REF: PCT Int. Appl., 2004052899, 24 Jun 2004

CON: STAGE(1) 5 minutes, 0 deg C; 15 minutes, 0 deg C
STAGE(2) 10 minutes; 40 minutes, -5 deg C
STAGE(3) 0 deg C

L3 ANSWER 4 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

RX(3) OF 71

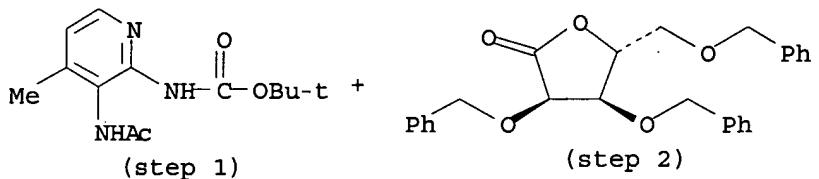


REF: Chemistry--A European Journal, 9(23), 5888-5898, 2003

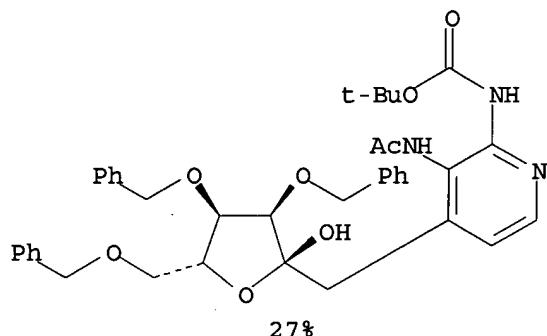
NOTE: stereoselective, 20:80 alpha:beta
 CON: STAGE(1) 20 minutes, -70 deg C
 STAGE(2) 10 minutes, -70 deg C; 1 hour, -70 deg C -> -40 deg C

L3 ANSWER 5 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

RX(1) OF 23



1. BuLi, THF, Hexane
 3. NH4Cl, Water



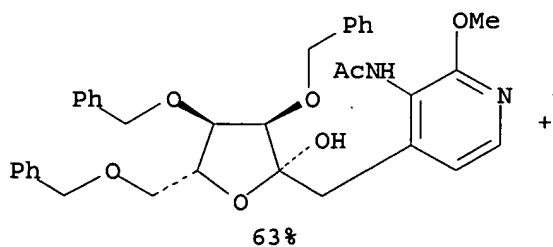
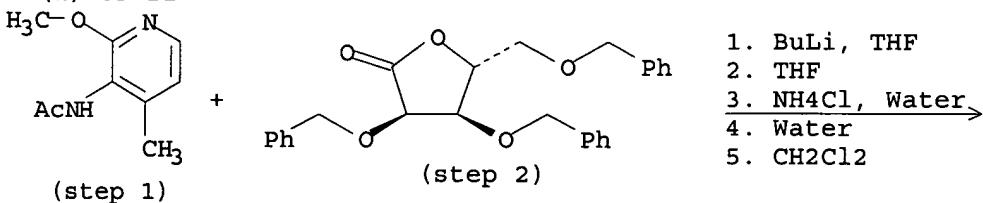
REF: Journal of Organic Chemistry, 68(16), 6466-6469; 2003

NOTE: stereoselective, other product also detected

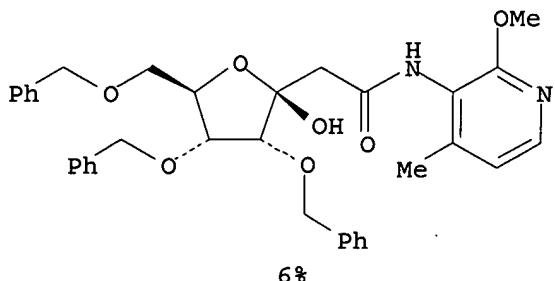
CON: STAGE(1) 15 minutes, -78 deg C; 20 minutes, -78 deg C -> 5 deg C;
 5 deg C -> -78 deg C
 STAGE(2) 15 minutes, -78 deg C; 5 hours, room temperature

L3 ANSWER 6 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

RX(1) OF 21



RX(1) OF 21



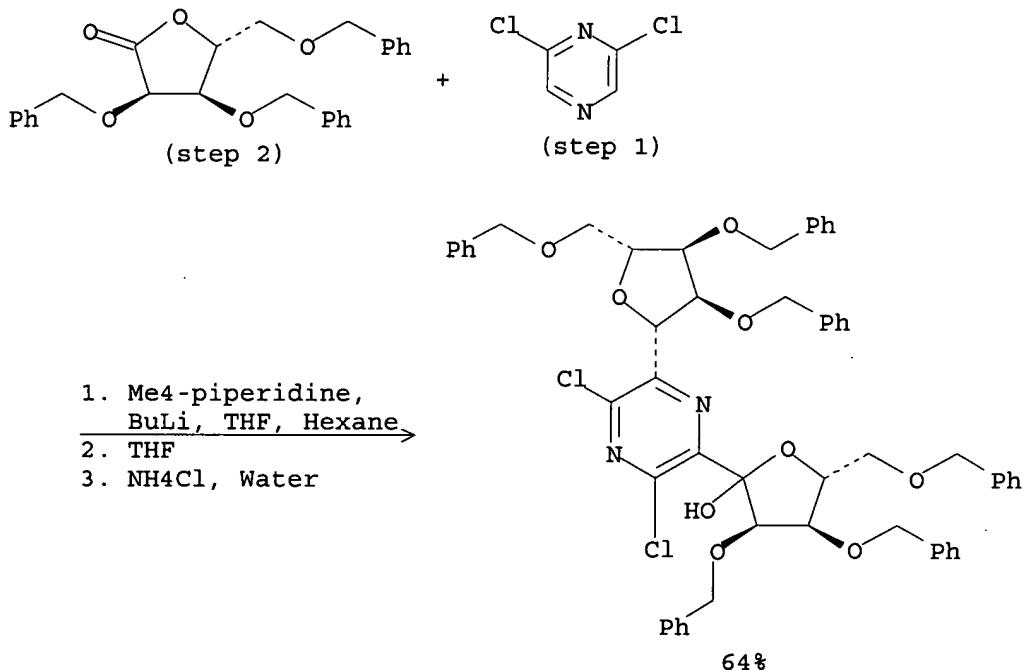
REF: *Synlett*, (9), 1479-1482; 2002

NOTE: stereoselective

CON: STAGE(1) 15 minutes, -78 deg C; 1 hour, -40 deg C
STAGE(2) 1 hour, -78 deg C

L3 ANSWER 7 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

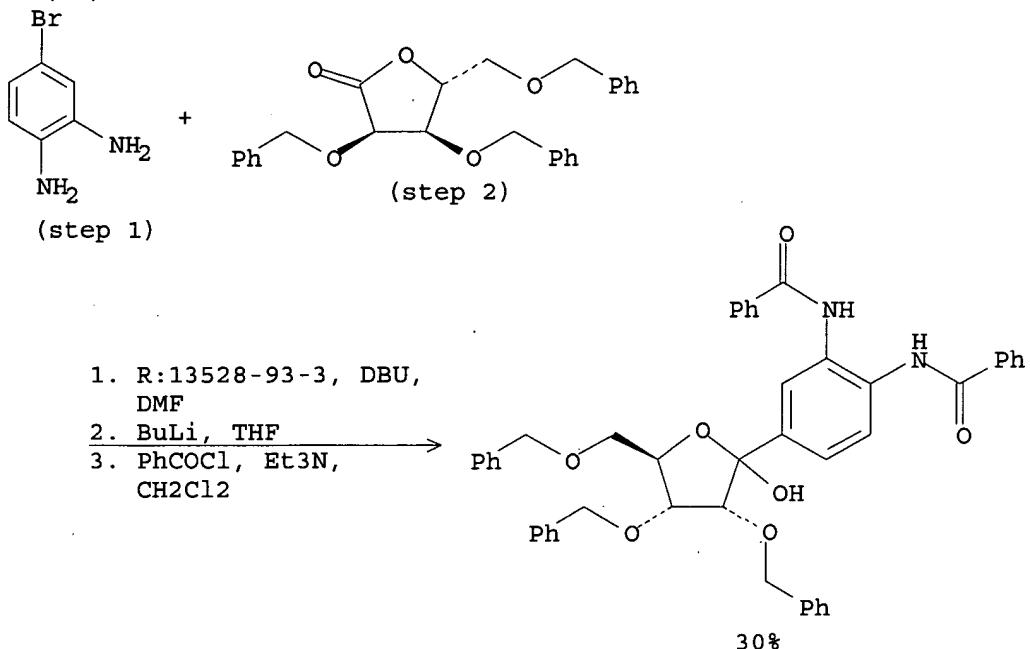
RX(1) OF 24



REF: *Journal of Organic Chemistry*, 66(14), 4783-4786; 2001

L3 ANSWER 8 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

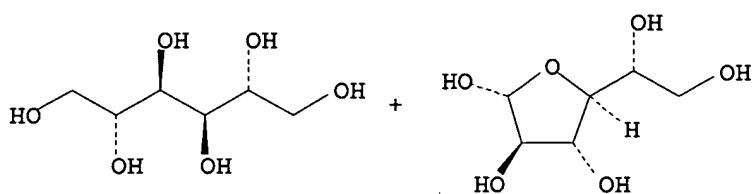
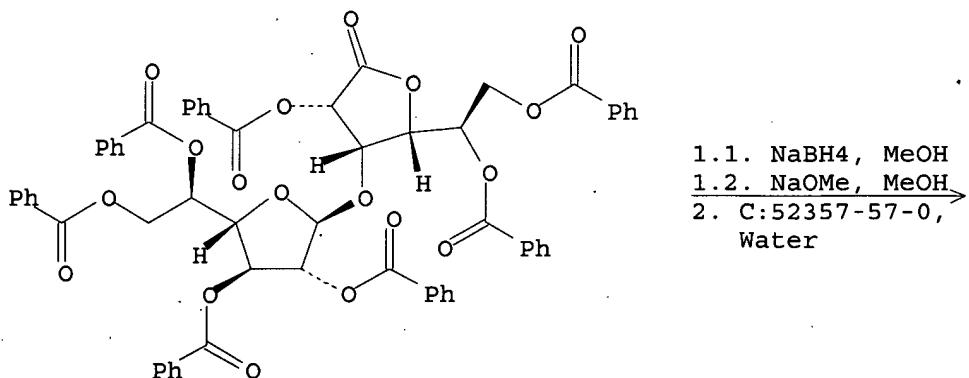
RX(11) OF 87



REF: Bulletin of the Chemical Society of Japan, 73(9), 1945-1954;
2000

L3 ANSWER 9 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

RX(15) OF 39 - 2 STEPS



REF: Carbohydrate Research, 311(4), 183-189; 1998

NOTE: 2) stereoselective, enzymic, biotransformation, sodium acetate buffered solution used, beta-D-furanosidase from *Penicillium fellutanum* used

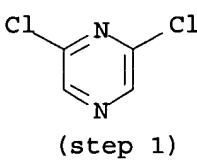
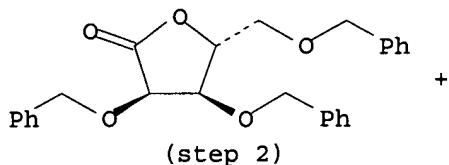
CON: STEP(1.1) overnight, room temperature

STEP(1.2) 3 hours, room temperature

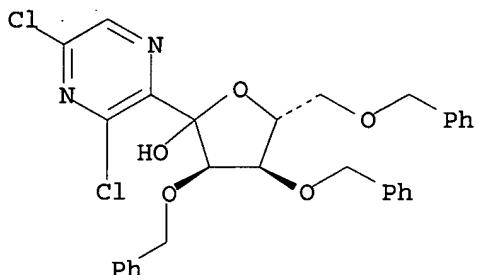
STEP(2.1) 1.5 hours, 37 deg C, pH 4; 2 minutes, 80 deg C

L3 ANSWER 10 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

RX(2) OF 3



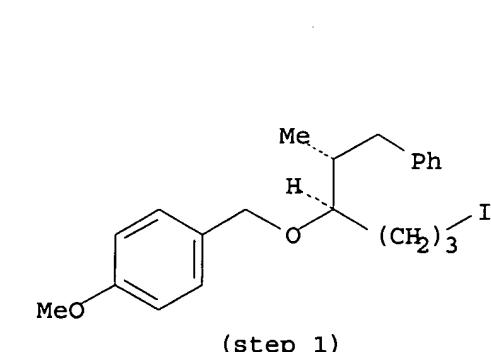
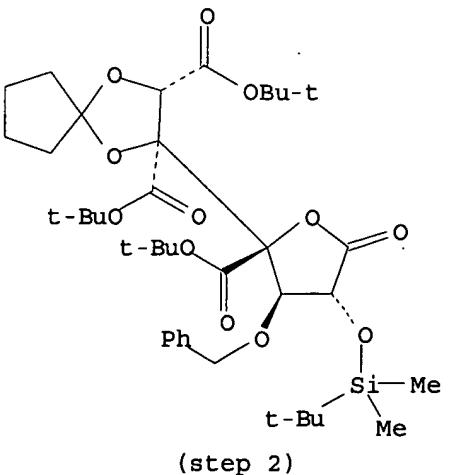
1. LiN(Pr-i)2, THF
2. THF
3. BF3-Et2O, Et3SiH,
CH2Cl2



REF: *Tetrahedron Letters*, 37(30), 5324-5328; 1996
NOTE: key step

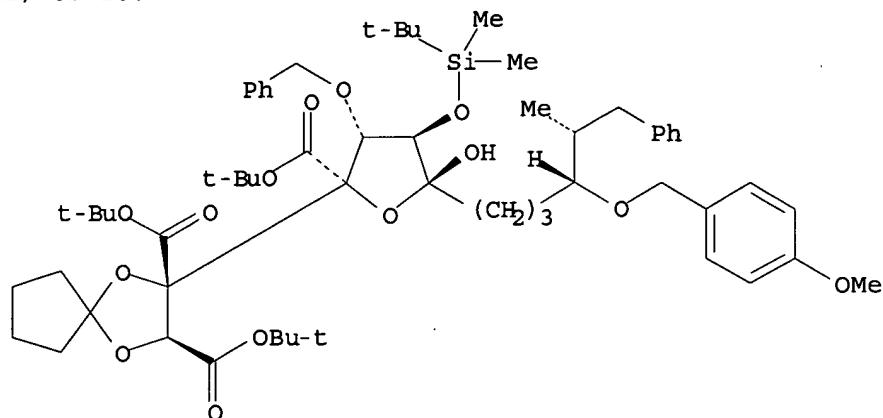
L3 ANSWER 11 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

RX(11) OF 180



1. t-BuLi, Hexane,
Et2O

RX(11) OF 180

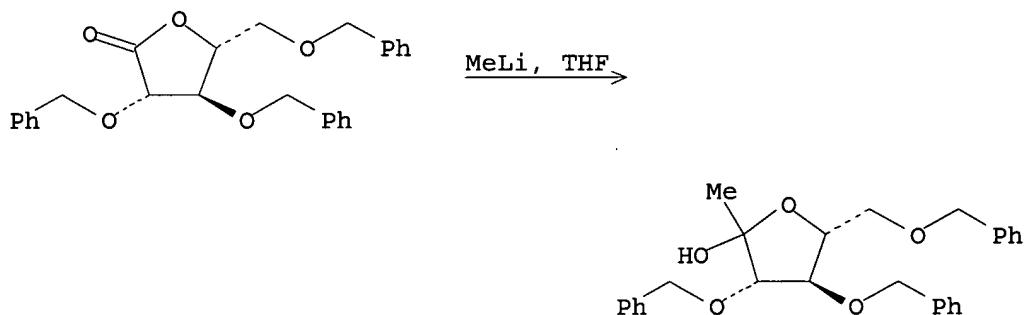


REF: Journal of the American Chemical Society, 116(26), 12111-12;
1994

NOTE: stereoselective

L3 ANSWER 12 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

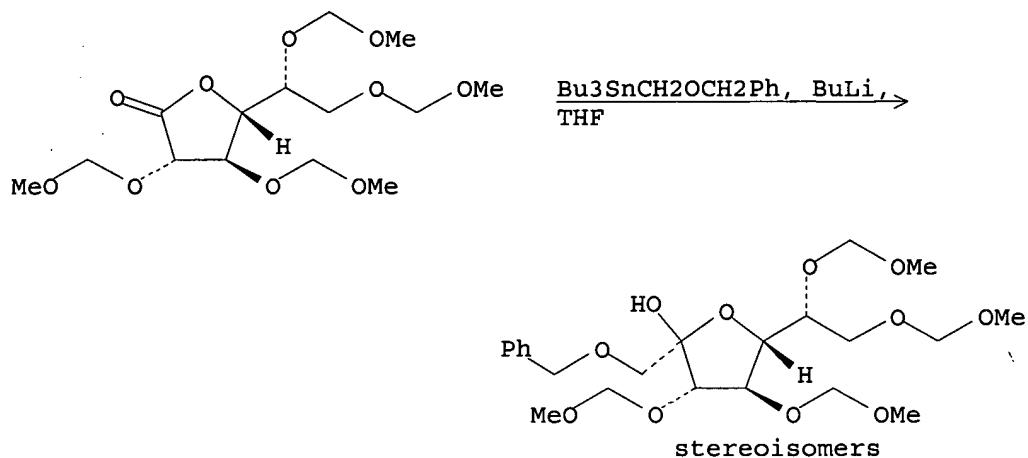
RX(2) OF 33



REF: Journal of the Chemical Society, Perkin Transactions 1: Organic and Bio-Organic Chemistry, (5), 517-18; 1995

L3 ANSWER 13 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

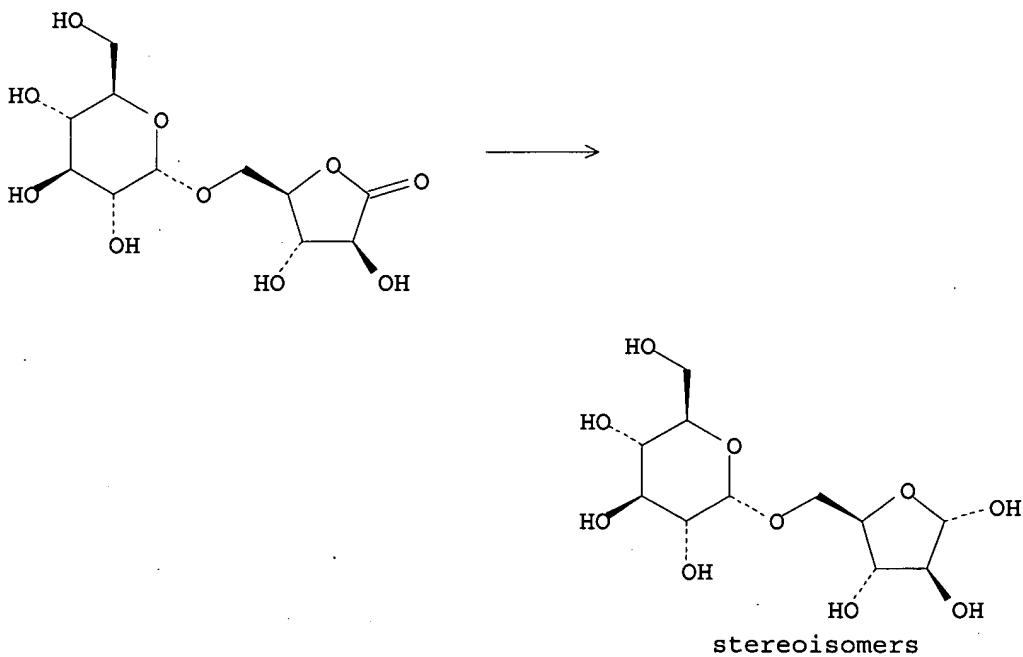
RX (4) OF 22



REF: Carbohydrate Research, 253,, 195-206; 1994
NOTE: KEY STEP; 81% OVERALL

L3 ANSWER 14 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

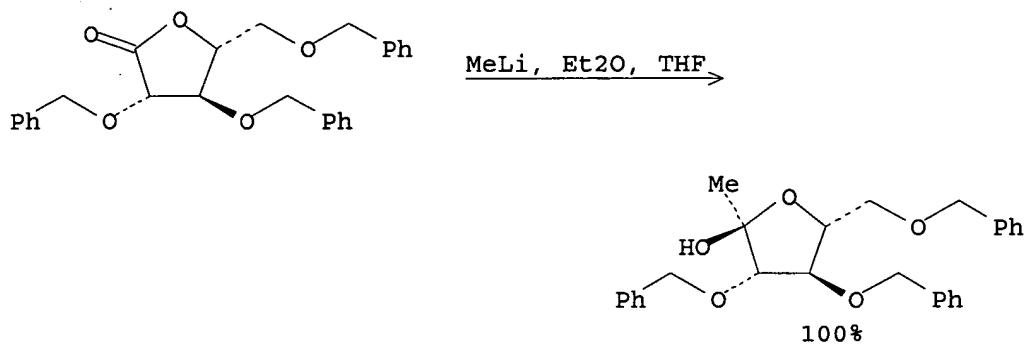
RX (3) OF 6



REF: Liebigs Annalen der Chemie, (9), 975-80; 1993
NOTE: 2 steps

L3 ANSWER 15 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

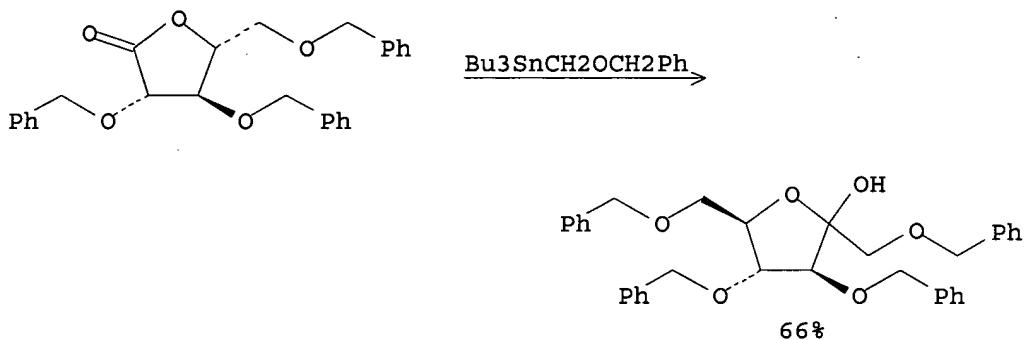
RX(5) OF 10



REF: Journal of Organic Chemistry, 57(4), 1304-6; 1992

L3 ANSWER 16 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

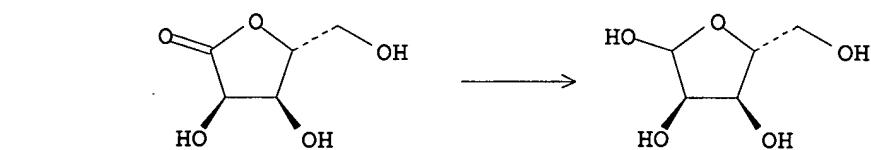
RX(1) OF 1



REF: Carbohydrate Research, 214(1), 187-92; 1991

L3 ANSWER 17 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

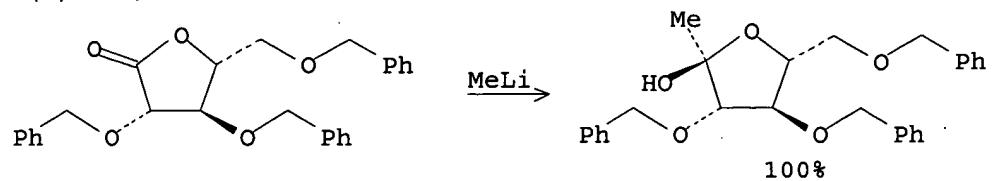
RX(1) OF 1



REF: Pol., 134957, 25 Jul 1986

L3 ANSWER 18 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

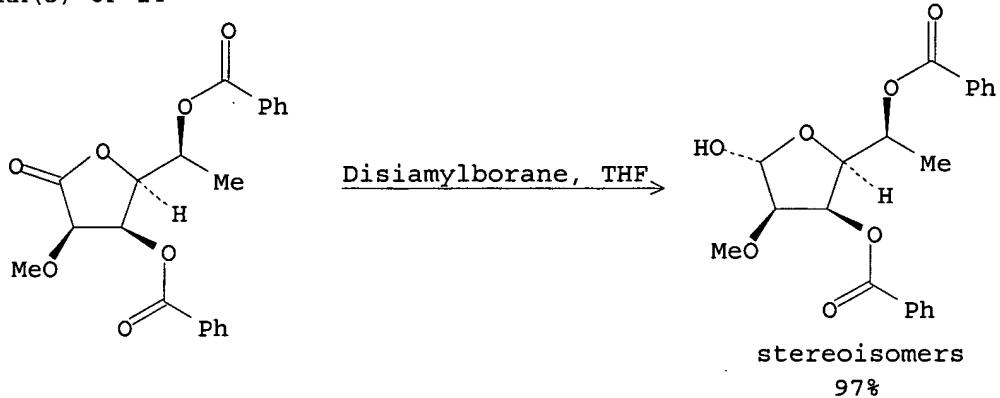
RX(1) OF 9



REF: Journal of the Chemical Society, Chemical Communications, (16), 1085-6; 1989

L3 ANSWER 19 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

RX(5) OF 14



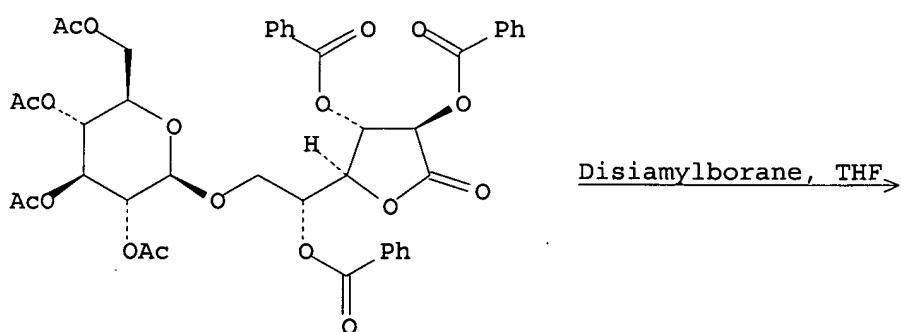
REF: Carbohydrate Research, 191(1), 130-7; 1989

L3 ANSWER 20 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

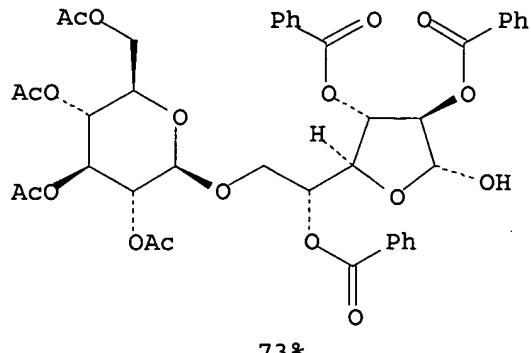
RX(13) OF 36 - REACTION DIAGRAM NOT AVAILABLE

L3 ANSWER 21 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

RX(6) OF 21



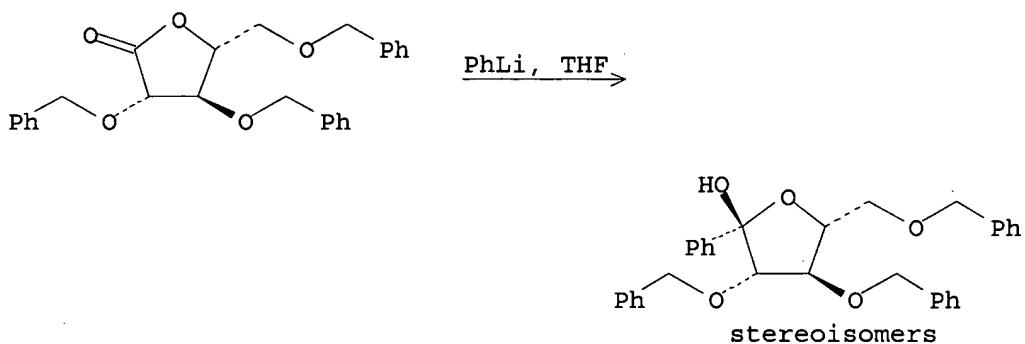
RX(6) OF 21



REF: Carbohydrate Research, 189, 79-86; 1989

L3 ANSWER 22 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

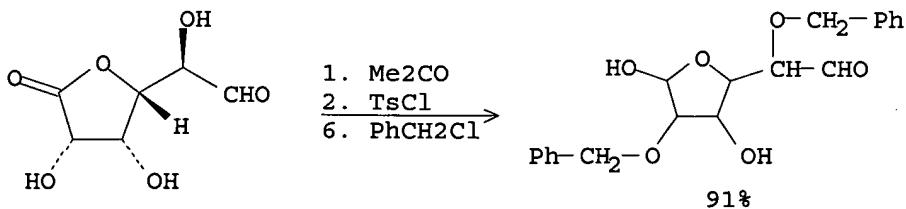
RX(7) OF 24



REF: Journal of Organic Chemistry, 54(3), 610-12; 1989

L3 ANSWER 23 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

RX(82) OF 183 - 7 STEPS

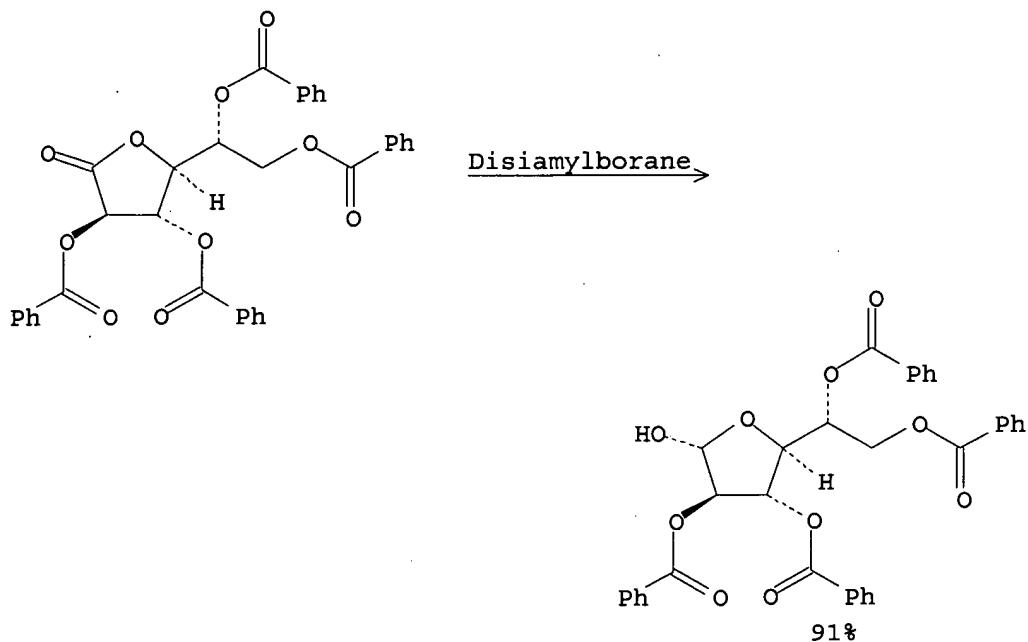


REF: Chemistry Letters, (1), 123-6; 1987

NOTE: 3) 88% overall

L3 ANSWER 24 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

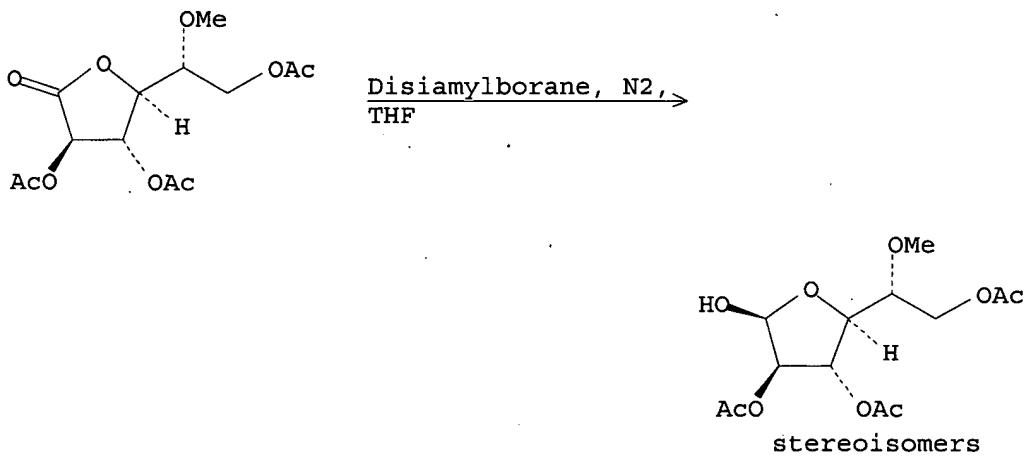
RX(1) OF 17



REF: Carbohydrate Research, 155, 247-51; 1986

L3 ANSWER 25 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

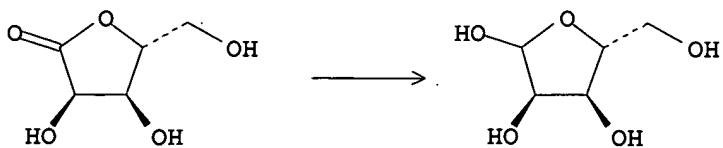
RX(5) OF 20



REF: Carbohydrate Research, 146(2), 233-40; 1986

L3 ANSWER 26 OF 26 CASREACT COPYRIGHT 2006 ACS on STN

RX(1) OF 3



REF: Pol., 121507, 30 Nov 1983

=> dis hist

(FILE 'HOME' ENTERED AT 10:45:43 ON 01 DEC 2006)

FILE 'CASREACT' ENTERED AT 10:46:04 ON 01 DEC 2006

L1	STRUCTURE UPLOADED
L2	4 S L1 SSS SAM
L3	26 S L1 SSS FULL
L4	0 S L3 AND (NAHTE OR SMI2)
L5	0 S L3 AND TELLUROHYDRIDE
L6	0 S L3 AND (SAMARIUM(A) IODIDE)
L7	0 S L3 AND (HYDROGEN(A) PALLADIUM(A) PHOSPHINE)
L8	0 S L3 AND (HYDROGEN(A) CATALYST)
L9	0 S L3 AND (ALUMINUM(W) T-BUTOXY(W) HYDRIDE)

$[\alpha]^{20}_D +34^\circ$ (5) and m.p. 145° (?). Various crystalline salts have been prepared (1), but they do not always give distinguishing melting points.

References

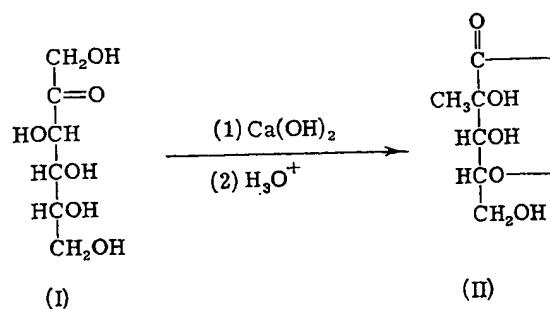
- (1) See J. C. Sowden, *Advances in Carbohydrate Chem.*, **12**, 35 (1957).
- (2) H. Kiliani, *Ber.*, **16**, 2625 (1883).
- (3) H. Kiliani and H. Sanda, *Ber.*, **26**, 1649 (1893).
- (4) H. Kiliani and P. Loeffler, *Ber.*, **37**, 1196 (1904); H. Kiliani and H. Naegeli, *Ber.*, **35**, 3528 (1902); H. Kiliani, *Ber.*, **44**, 109 (1911).
- (5) J. U. Nef, *Ann.*, **376**, 1 (1910).
- (6) L. M. Utkin and G. O. Grabilina, *Doklady Akad. Nauk S.S.R.*, **93**, 301 (1953).
- (7) H. Kiliani and F. Eisenlohr, *Ber.*, **42**, 2603 (1909).

[118] " α "-D-Glucosaccharino-1,4-lactone

2-C-Methyl-D-ribo-pentono-1,4-lactone from D-Fructose

BY ROY L. WHISTLER AND J. N. BEMILLER

Department of Biochemistry, Purdue University, Lafayette, Indiana



Introduction

" α "-D-Glucosaccharino-1,4-lactone (2-C-methyl-D-ribo-pentono-1,4-lactone) (II) (1) is prepared by the action of calcium hydroxide on D-fructose (I) (2) or "inverted" sucrose (3, 4). The yields in either case are about the same.

Procedure

To a solution of 100 g. of β -D-fructose¹ (I) in 1 liter of boiled water is added 10 g. of calcium hydroxide. The mixture is flushed with nitrogen (footnote 1, Vol. II [115]) and kept 14 days at room temperature with frequent shaking, after which time an additional 40 g. of calcium hydroxide is

¹ "Inverted" sucrose may also be used (3, 4).

added. The mixture is again flushed with nitrogen and kept 6-8 weeks at room temperature with occasional shaking.² The mixture is filtered, and the filtrate is saturated with carbon dioxide and filtered again. A concentrated aqueous solution of 38 g. of oxalic acid dihydrate³ is then added with vigorous stirring. The mixture is warmed on a steam bath and filtered. The remaining calcium ions are removed by passing the filtrate through a column of 175 ml. of Amberlite IR-120(H⁺). The column is washed until the effluent is neutral. The decationized solution and washings are concentrated under reduced pressure to a thin syrup which is placed in a refrigerator for crystallization.⁴ After several days, the crystals of "α"-D-glucosaccharino-1,4-lactone (II) are filtered from the mother liquor and recrystallized from water; yield about 10 g., m.p. 160-161°, [α]₂₀^D +93° (water) (2, 5).

Derivatives

Reported derivatives include the phenylhydrazide (5, 6) m.p. 167-169°, [α]₂₀^D +50° (water), the "anilide" (7), m.p. 193-195°, [α]₂₀^D +58° (water), [α]₂₅^D +55° (95% ethanol), and the anhydrobenzimidazole, 2-(1,4-anhydro-1-C-methyl-D-ribo-tetrahydroxybutyl)benzimidazole (8), m.p. 240-241°. The crystalline mono-O-isopropylidine derivative has also been prepared (9). Various crystalline salts have been reported (4), but they do not always give distinguishing melting points.

References

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- (3) H. Kiliani, *Ber.*, **15**, 701, 2953 (1882); *Ann.*, **218**, 361 (1883).
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- (5) J. U. Nef, *Ann.*, **376**, 1 (1910).
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- (8) J. C. Sowden and D. J. Kuenne, *J. Am. Chem. Soc.*, **75**, 2788 (1953).
- (9) L. M. Utkin and G. O. Grabilina, *Doklady Akad. Nauk S.S.R.*, **93**, 301 (1953); J. C. Sowden, M. G. Blair, and D. J. Kuenne, *J. Am. Chem. Soc.*, **79**, 6450 (1957).

² The long period of standing at room temperature can be replaced by several-hours heating at 100° (2a), but the yield obtained by this method is unsatisfactory (3).

³ An amount of oxalic acid which is about 90% of that required to remove all the calcium ions in the filtrate should be added. This amount may vary and should be determined on an aliquot of the filtrate for each preparation.

⁴ Frequently, seed crystals are needed. These may be obtained by extracting a portion of the syrup in a continuous extractor for 24 hr., concentrating the extract to a sirup, and crystallizing the sirup from ethyl acetate by the addition of petroleum ether (30-60°).

SHEET 1/6

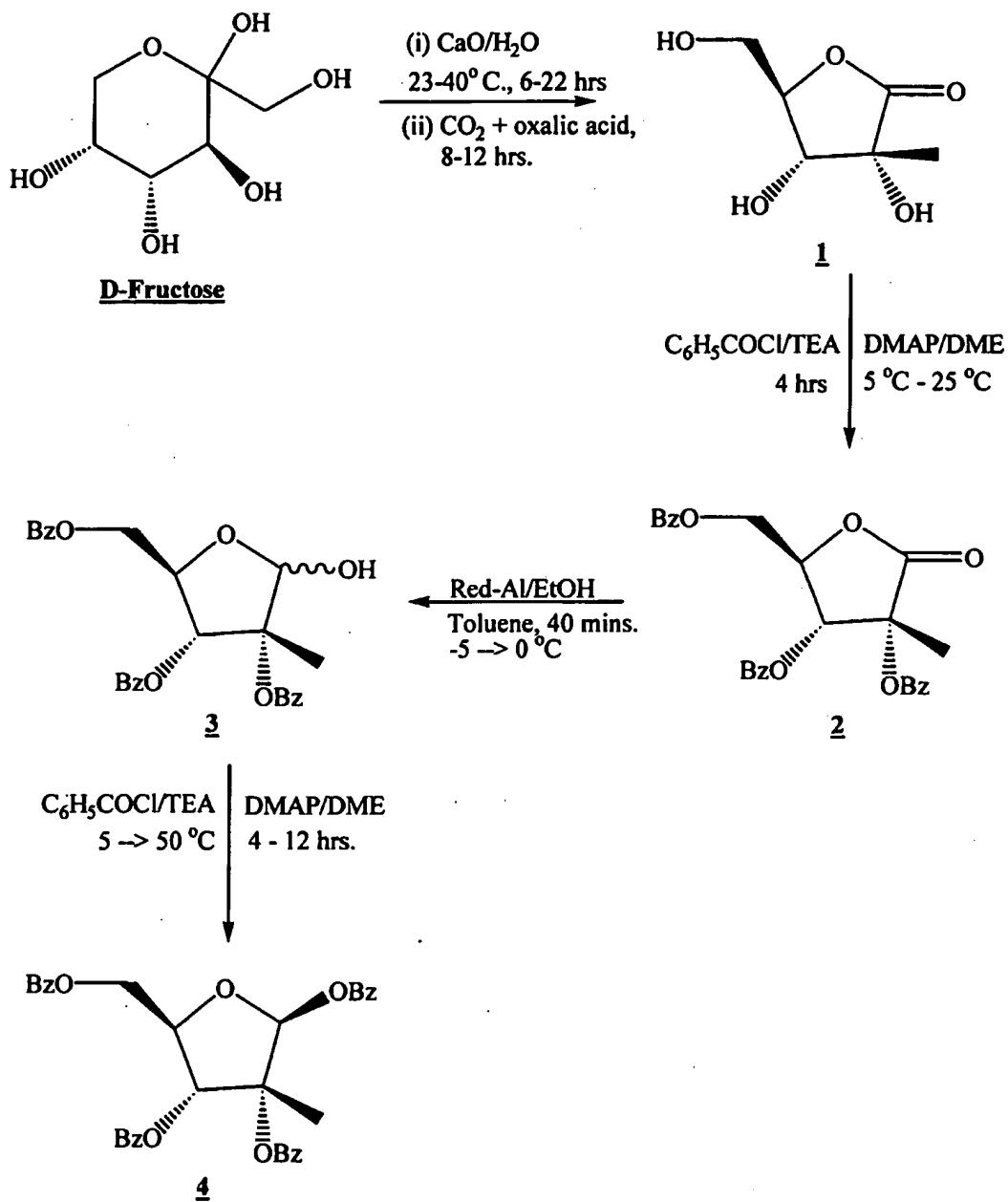


FIGURE 1

SHEET 2/6

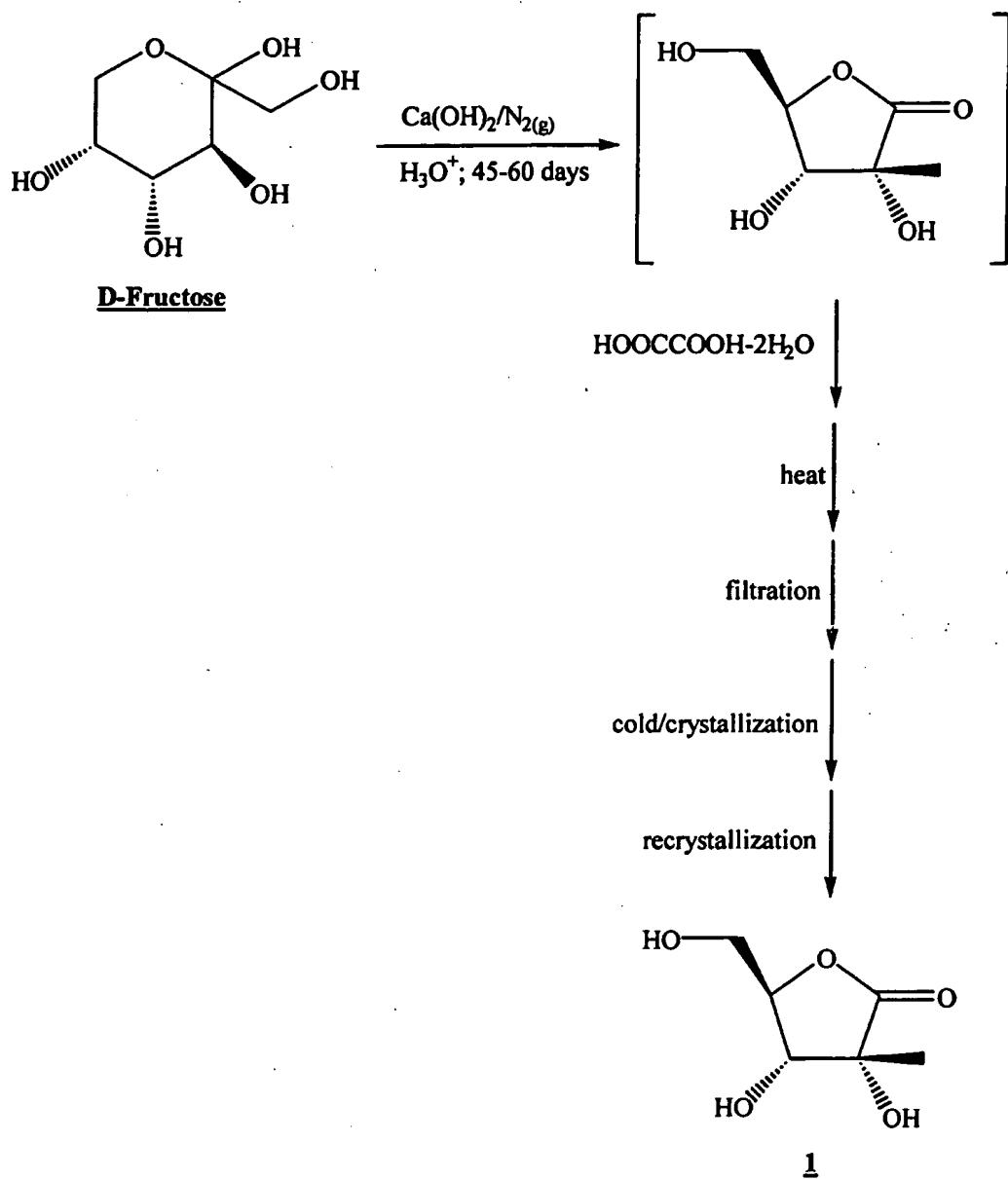
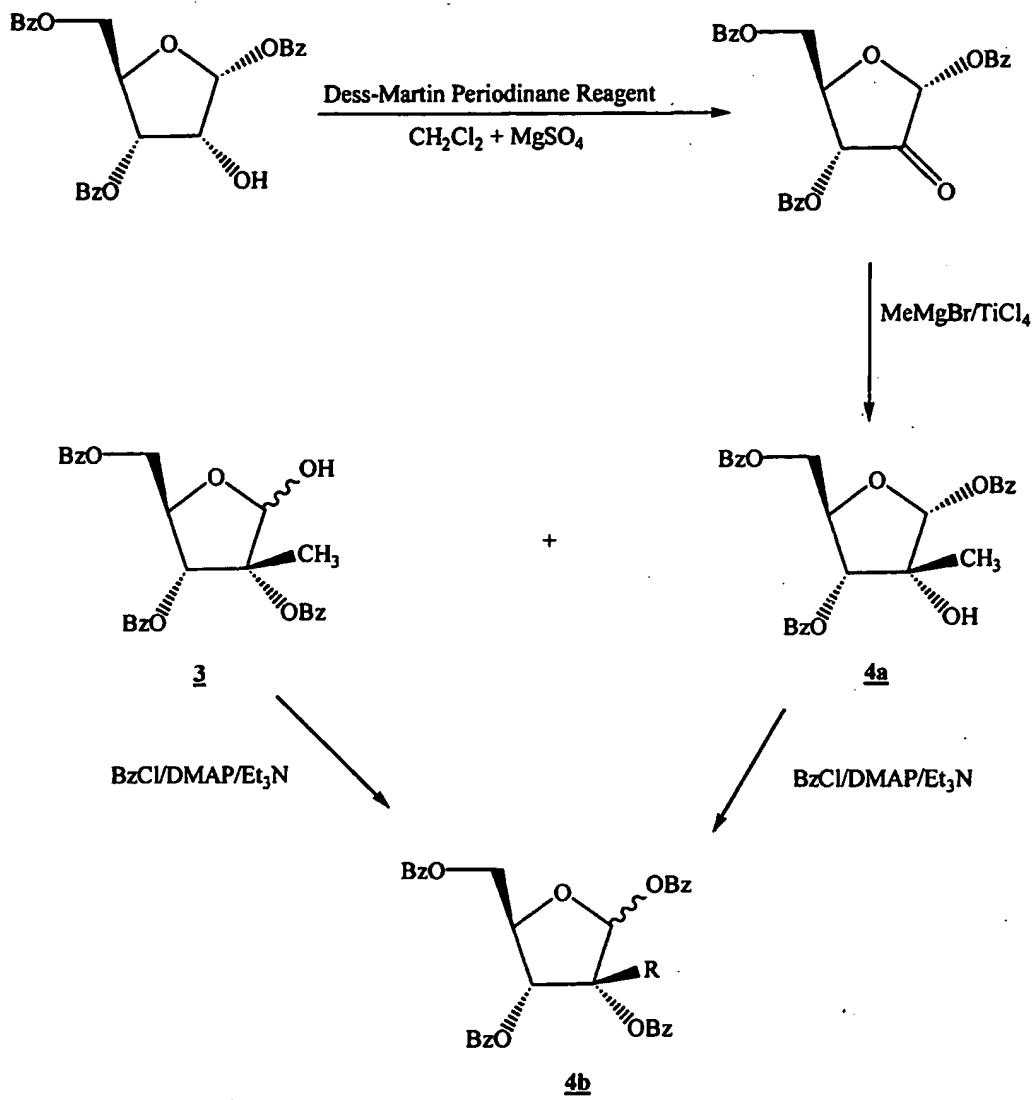


FIGURE 2

SHEET 3/6



Taken from Harry-O'kuru et al., J. Org. Chem., 1997, 62(6):1754-59.

FIGURE 3

SHEET 4/6

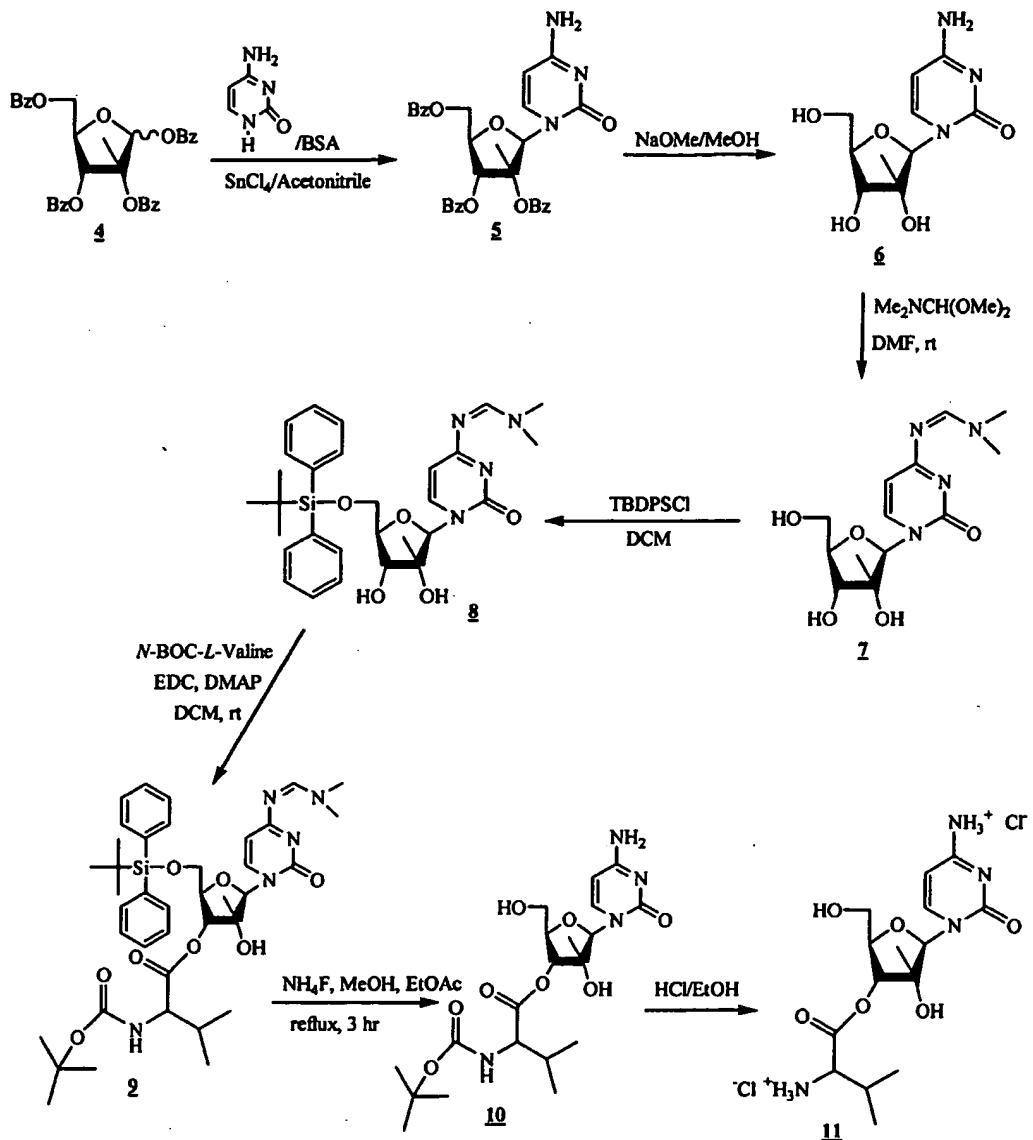


FIGURE 4

SHEET 5/6

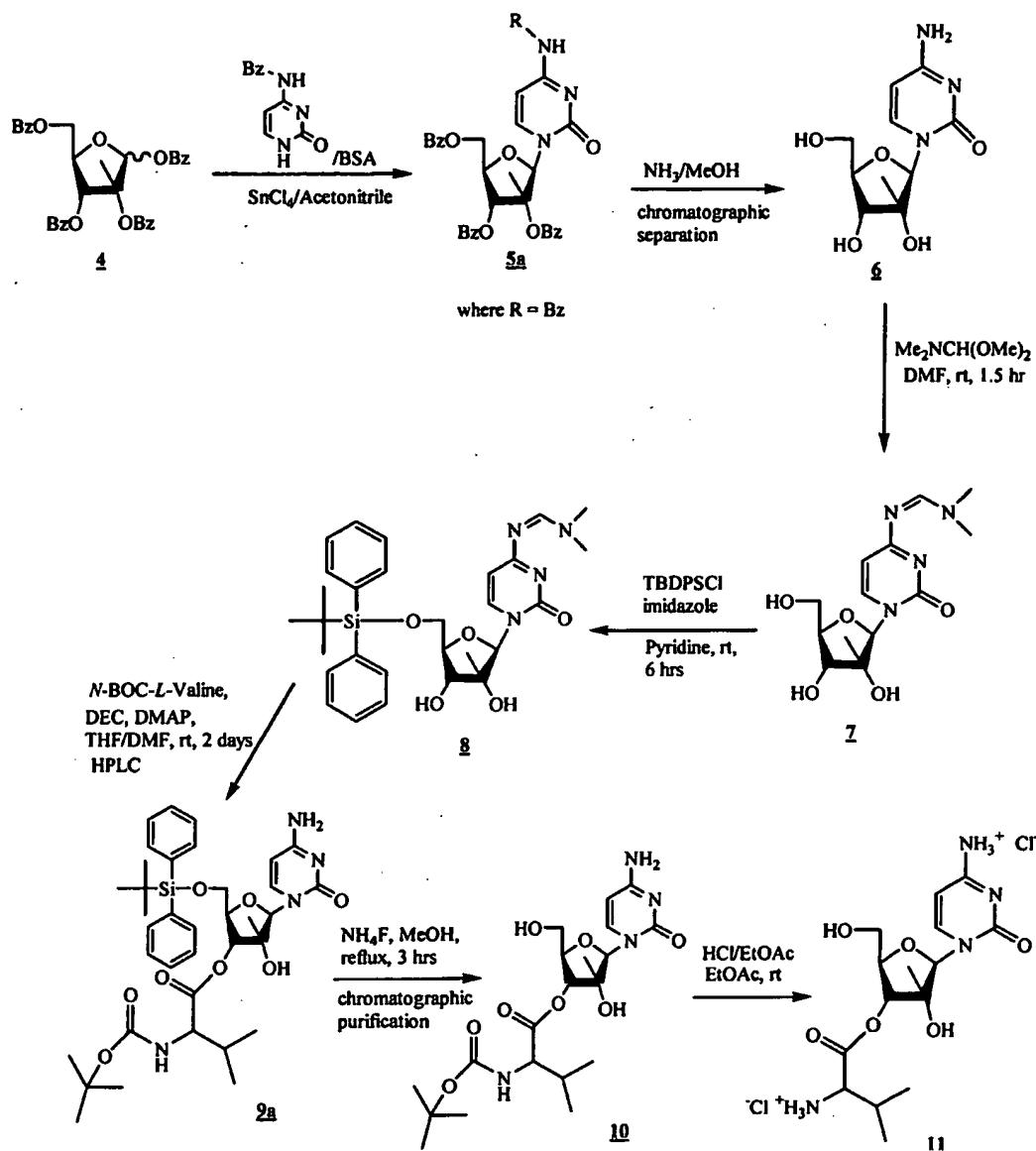


FIGURE 5

SHEET 6/6

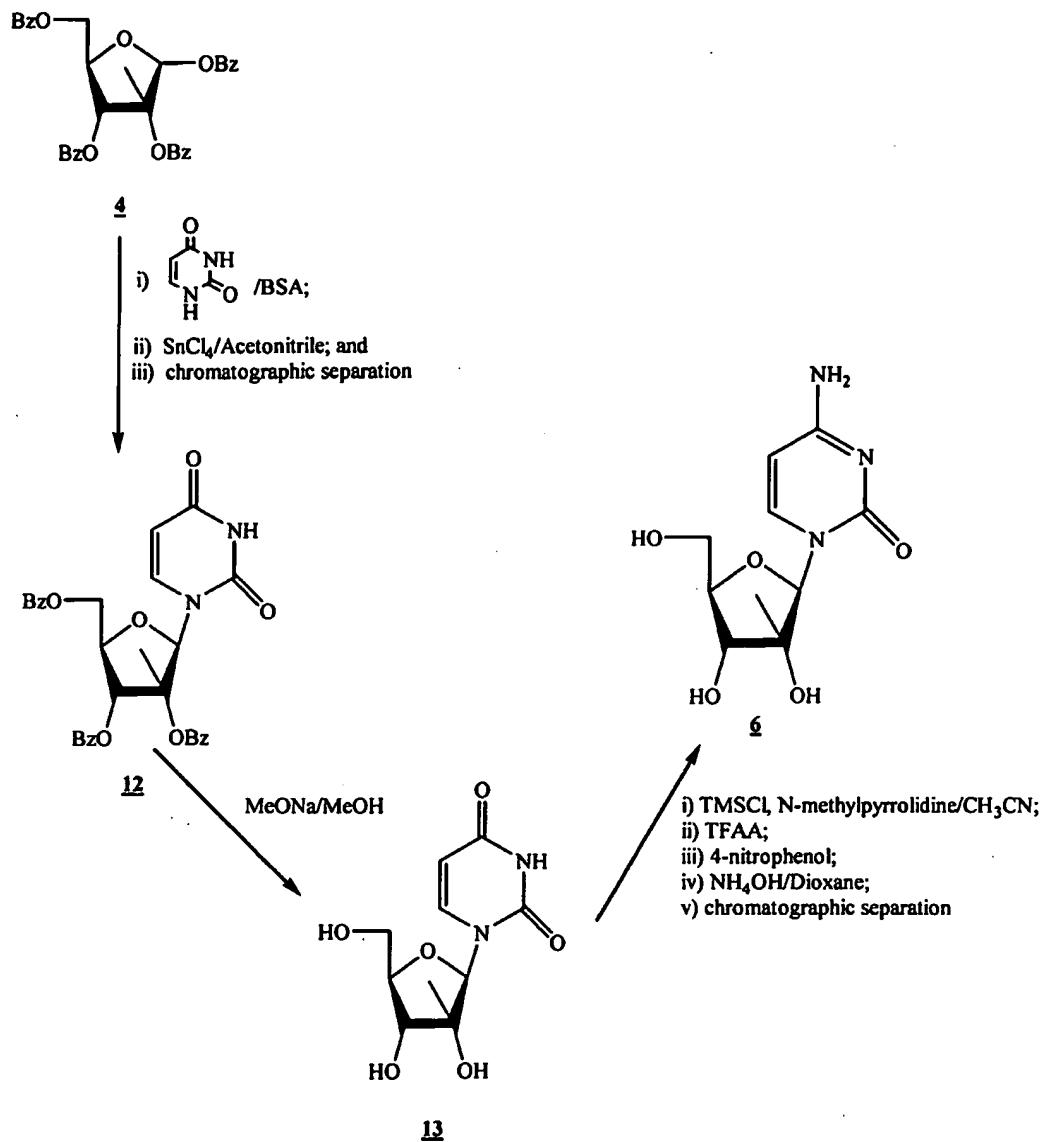


FIGURE 6